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OVERVIEW

Summary of Test Methods

The following protocols for extractions and interpretation of flammable liquid evidence in the Alaska Department of Public Safety Crime Detection Laboratory use the following ASTM (American Society of Testing Materials) standards as references:

- E 1386-00 Standard Practice for Separation and Concentration of Ignitable Liquid Residue from Fire Debris Samples by Solvent Extraction
- E 1388-00 Standard Practice for Sampling of Headspace Vapors from Fire Debris Samples
- E 1412-00 Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris by Passive Headspace Concentration
- E1618-10 Standard Test Methods for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry
- E 1387-01 Ignitable Liquid Classification System

The method utilizes a gas chromatograph (GC) which is interfaced to a mass spectrometer (MS) with a data system capable of storing and handling chromatographic and mass spectral data (ChemStation™).

The total ion chromatograms (TIC) and extracted target ion chromatograms are evaluated by visual pattern matching against known standards for the purpose of detecting and identifying ignitable liquid residues. Specific ions are indicators of certain classes of compounds, so extracted ion profiles from ASTM

- E 1618-94 are included for guidance in standard to sample comparisons.

Significance and Use

The identification of an ignitable liquid residue in samples from a fire scene can support the field investigator's opinion regarding the origin, fuel load, and incendiary nature of the fire.

The identification of an ignitable liquid residue in a fire scene does not necessarily lead to the conclusion that a fire was criminal in nature. Further investigation may reveal a legitimate reason for the presence of ignitable liquid residues.

Due to the volatility of ignitable liquids and variations in sampling techniques, the absence of detectable quantities of ignitable liquid residues does not necessarily lead to the conclusion that ignitable liquids were not present at the fire scene.

Materials normally found in buildings, upon exposure to the heat of a fire, will form pyrolysis and combustion products. Extracted ion profiling and target compound identification techniques described may facilitate the identification of an ignitable liquid in the extract by reducing interference by components generated as products of pyrolysis.

EVIDENCE COLLECTION

Fire Debris evidence should be collected and sent to the laboratory in paint cans or fire debris bags. It is important to collect the evidence as soon as possible and place into an air tight container. Use lined metal paint cans (the interior has a coating, usually grey, Sherwin Williams or NAPA) or Ampac™ fire debris bags. Collection containers are regularly checked with new lot numbers to assure that no contamination is present to interfere with the analysis.

Fill up to 75% or less of the capacity of the container to leave room for air space. Use optimal size cans that match the amount of evidence. Ideally place a hand full of debris in a quart size can and a half of a shovel full of debris in a gallon can.

If liquids are to be sent to the laboratory as evidence, place half of a teaspoon of liquid into a can. Another options for liquids is a new leak proof vial, tightly sealed, that contains approximately 2 mL of liquid.

Send only a representative sample in to the lab. Safely secure the remaining liquid in an area not common to other items of evidence.

Change gloves between each sample. Do not include gloves and clean tools between samples.

Collect a comparison sample (control sample) of similar debris away from the target area collected. Example: a portion of carpet in the corner of room which is not suspected of having an accelerant.

INSTRUMENTATION, EQUIPMENT, & MATERIALS

1. Gas Chromatograph Mass Spectrometer (GCMS)
   This laboratory utilizes:
   - Hewlett Packard Gas Chromatograph model 6890 with a 7638 series autosampler
   - Hewlett Packard 5973 mass spectrometer (MOE)
   - Approximately 30 meter x 0.25 mm HP-1ms fused silica capillary column coated with 100% methyl silicone film thickness of 0.25 µm (a phenylmethyl silicone column such as a HP-5ms may also be used.)
   - Mass spectrometer capable of scanning from 40-400 m/e.
   - ChemStation software with ability to retrieve mass spectral data and compare to a library
   - Carrier gas: chromatography grade helium
2. A reference ignitable liquid collection
3. Oven
4. Reagent grade (or better) carbon disulfide and pentane
5. Charcoal adsorption strips
   These polymer strips are used to adsorb organic vapors and are available from Albrayco Laboratories.
6. Other consumables: paint cans, fire debris bags, vials, inserts, caps, pipets, paper clips, magnets, razor blades, and thread

QUALITY CONTROL

1. **Calibration**
   The MS is tuned at least once per month that the instrument is in use. Records are maintained in the LIMS: CS INST 2011 (or current year).
2. **Standard**
   SAM is run with every case (or batch of cases).
   Standard Accelerant Mixture **(SAM)**
   Stock: equal parts gasoline, kerosene, and diesel fuel
   Working: 5% Stock diluted with carbon disulfide (C_2S)
3. **Test Mixtures**
   These are run several times a year to test the sensitivity and resolution of the component peaks. Results are recorded in LIMS.
   Cerilliant Resolution Test Mixture (catalogue number ERR-002)
   Restek E-1618-97 Test Mixture (catalogue number 31613)
4. A **blank sample** is generated with every new batch of fire debris evidence utilizing an unused activated charcoal strip (c-strip), new vial, new insert, and

carbon disulfide. A previous 'blank' paint can, a new paint can, or a new fire debris bag is used for the extraction. This blank sample is analyzed before all casework samples on the GCMS.

Instrument Quality Control documentation is maintained and archived in the GCMS Logbook for MOE and/or the laboratory's LIMS (Justice Trax).

Chemical and Equipment Quality Control is maintained in the Fire Debris Quality Control Logbook and/or the laboratory's LIMS.

The MS is periodically cleaned and maintained (source, septa, liner etc.). See instrument logbook.

The syringes are cleaned thoroughly between injections.

Blank sample analyses are documented in the individual case files.

Every case file includes a standard chromatogram, either the 5% SAM or an appropriate standard from the Reference Ignitable Liquid Collection. The standard chromatograms are run under the same chromatographic conditions as those used to produce the evidence chromatogram.

At least one standard and an appropriate blank are included in every sequence.

Activated charcoal strips (c-strips), carbon disulfide, fire debris bags, pentane, razor blades, paper clips, magnets etc. are purchased and stored to ensure they are free of extractable hydrocarbons. Additionally, new lot numbers of carbon strips and carbon disulfide are tested before use to insure that no contamination is present.

1. One representative adsorbent charcoal strip from each new lot should be tested in the same manner as an actual case to insure the quality of the products adsorption efficiency and the lack of contaminants. Documentation will be maintained in the Fire Debris Quality Assurance logbook.
2. The adsorption efficiency of the activated carbon strip is periodically checked by running the passive headspace extraction procedure on a known volume of SAM.
3. The solvent purity is checked by evaporating the carbon disulfide to at least half the original volume. The evaporated solvent is analyzed by GCMS and documented in the Fire Debris Quality Assurance log book.

A negative carbon strip extraction control is included with each batch of samples to show that no cross-contamination has occurred. This blank control is analyzed before each evidence sample and documentation is found in the case file. Significant amounts of contaminants in the negative control need to be investigated and documented prior to the issuance of a report. Repeating the negative control may be necessary. Results will not be reported until any contamination issues have been resolved.

The temperature of the oven used should be 65ºC plus or minus 15º. This is periodically monitored. Records are not maintained.

Certified ignitable liquid standards are not necessary. Most reference ignitable liquids can be obtained from commercial and retail sources.

Separation and Concentration Procedures

Passive Headspace Concentration

This procedure is useful for removing small quantities of ignitable liquid residue from samples of fire debris. The method is sensitive and essentially nondestructive. It is the method of choice for arson samples that are packaged in airtight containers (i.e. paint cans, fire debris bags, and jars). Passive Headspace Concentration may not distinguish between #1 and #2 fuel oils. It also may not detect light oxygenated solvents.

1) Briefly open and examine the fire debris sample in order to determine that it is consistent with its description. Resolve any discrepancies.
2) Place an adsorbent charcoal strip (typically a full size strip 10 x 20 mm) on the end of a paper clip and suspend in the evidence container. The paper clip may be attached to the inside of the lid by use of a magnet or hung in the can by a piece of thread.
3) Reseal the container and place in an oven set to approximately 65° C overnight. Extraction temperature and time can vary from 50° to 80° C and 2 to 24 hours. Longer times or higher temperatures are required for the adsorption of higher boiling point compounds, dense sample matrices, or for adsorption of very small quantities of volatile hydrocarbons. The adsorption temperature and duration may vary based on the sample. When other evidentiary considerations arise, such as document or latent print examinations, it may be appropriate to conduct the adsorption at ambient temperature (approximately 20° to 25° C) for extended periods

(24 hours or longer) to minimize damage. Room temperature adsorption may also be appropriate to detect low molecular weight compounds.

4) After heating, the container is removed from the oven and is allowed to cool.
5) The charcoal strip is removed from the container and divided into two pieces. One portion is placed in an appropriately labeled vial and returned with the evidence. The other is placed into a labeled auto sampler vial with insert. The vial insert is filled with carbon disulfide and the vial sealed with a screw cap septum. The sample is now ready for instrumental analysis.
6) A parameter sheet containing the lot number and amount of the adsorbent, the approximate oven temperature, the approximate length of time in the oven, the elution solvent type, and approximate elution volume is included with the case file. Information is also included about the column and the GCMS parameters.

Solvent Extraction of fire debris or containers

Due to size and/or packaging, certain items may not be suited for using the passive adsorption technique. This method is useful for extracting ignitable liquid residues from very small or very large non-porous containers. It is also useful when attempting to distinguish between various grades of fuel oils.

1) Open and examine the fire debris sample in order to determine that it is consistent with its description. Resolve any discrepancies.
2) Place a representative sample in a clean beaker.
3) Thoroughly moisten and mix the sample with a sufficient volume of solvent such as pentane or carbon disulfide.
4) For some items of evidence (such as fuel cans) the solvent may be added directly to rinse any ignitable liquid residues.
5) Decant the solvent into a second clean beaker and filter if necessary.
6) Evaporate the eluent to a small volume (approximately 1 mL) to concentrate any ignitable liquid residues that may be present.
7) The sample is now ready to be placed in an autosampler vial for instrumental analysis. The elution extract is returned with the evidence after it is analyzed by GCMS.

Sampling of Head Space Vapors from Fire Debris Samples

This method is not routinely utilized. It may be employed when the investigator suspects that a light oxygenate is involved, an odor alcohol, ester, or ketone exists in the evidence, and 'negative' results are obtained using the passive headspace method.

This practice is the least sensitive of the sample preparation techniques and may not detect quantities of less than 10 µL of petroleum product. Because this separation takes place in a closed container, the sample remains in approximately the same condition in which it was submitted. Due to variables in the debris sample condition prior to headspace sampling, complete reproducibility of chromatograms may be difficult to obtain. High concentrations of highly volatile compounds may overwhelm the headspace, inhibiting the recovery of less volatile components.

1) Open and examine the fire debris sample in order to determine that it is consistent with its description. Resolve any discrepancies.
2) A gas-tight syringe blank consisting of air is run on the GCMS using an appropriate method.
3) Punch a small hole in the lid of the sample can with a tool such as an awl and immediately place a piece of tape over the hole.
4) Place the can in an oven at approximately 65 degrees C for about 30 minutes (if it is to be heated).
5) Remove the can from the oven and immediately insert the syringe needle through the taped hole, pump it several times, and withdraw approximately one milliliter of headspace sample. Inject into the GCMS using an appropriate method. The optimum sample size will vary with sample concentration.
6) Reseal the hole with tape.

Liquid Samples

If a liquid sample is received as an item of evidence, it may be injected into the GCMS after dilution of the sample with carbon disulfide at an approximate ratio of 1:100.

Flame Test

If a liquid sample is submitted, a flame test may be run. This may be accomplished by placing a few drops of the liquid into a non-flammable container such as metal weigh boat and then expose the liquid to a flame. The results of this test are then documented in the bench notes.

Instrumental Analysis (Gas Chromatography/Mass Spectrometry)

Utilizing one of the above described extraction techniques a sample is obtained for analysis by gas chromatography/mass spectrometry. The resulting total ion chromatogram, mass spectral data, and extracted ion profiles are then evaluated by comparison to known standard data.

GC parameters (Agilent 6890)

Injector	250° C
Initial Temperature:	40°
Initial Time	4 minutes
Rate	10°/minute
Final Temperature	280° (can be higher for heavier samples)
Final time	2 minutes
Total run time	30 minutes

MS parameters (Hewlett Packard 5973N MSD)

Inlet	GC
Acquisition mode	Scan
Low mass	31
High mass	350

Note: These are suggested conditions only. Other parameters may be used as long as suitable resolution is obtained. Questioned samples and known standards must be run using the same conditions.

Analysis setup:

1. On the workstation computer, select MSD icon.
2. From the menu bar select SEQUENCE.
3. Choose EDIT SAMPLE LOG TABLE and enter the appropriate information:
    - TYPE – sample, blank, or calibration standard
    - VIAL – position number in autosampler tray
    - METHOD – desired parameters (usually EMARSON1)

- SAMPLE NAME – lab number and item number, blank or sample

Repeat this process until all data is entered. Select OK and review the sequence table to ensure that all vials are logged correctly. Check the level of solvent vials on the autosampler, select SEQUENCE, then RUN.

Data

1. Access to data:
   - On the workstation computer select ENHANCED DATA ANALYSIS icon
   - From the menu bar select FILE
   - Choose LOAD DATA FILE and select desired data file
   - To confirm the identity of a peak on the chromatogram, double right click on the peak.
   - To show library matches, double right click on the mass spectrum.
   - Total ion chromatograms with mass spectral data as well as extracted ion profiles from evidence samples and standards are utilized in reporting decisions and are retained in the LIMS case file.
   - Raw data files are retained for technical review use.
2. Initial data analysis consists of a visual comparison of the total ion chromatogram to reference ignitable liquid chromatograms. The essential requirement for making a classification is noting points of correlation and similarities between the sample chromatogram and the reference ignitable liquid chromatogram obtained under the same conditions. Pattern matching requires that the entire pattern used for comparison be displayed at the same sensitivity. External libraries are intended only to give guidance for selection of reference ignitable liquids. The carbon number range is determined by comparing the chromatogram to a reference or test mixture containing known normal alkanes. Mass chromatography may be carried out using target compound analysis.
3. The mass spectrum of major peaks may be compared to the NIST98 or another suitable library.

Mass Chromatograms

The GC/MS Chemstation™ is used to produce total ion chromatograms as well as extract and draw extracted ion profiles for major ions characteristic of each compound type. Individual mass chromatogram ion profiles for two or more characteristic ions of the same functional groups or of similar magnitude may be

summed to enhance the signal-to-noise ratio and to decrease interference by extraneous compounds that contain only one of the ions or to create summed profiles characteristic of specific classes of hydrocarbons.

Mass chromatograms for an unknown sample are compared against the corresponding mass chromatograms from reference material samples. This is generally done by visual pattern recognition.

Major peaks in the mass chromatograms should be identified by searching their mass spectra against a suitable library. The final identification must be made by the analyst on the basis of the mass spectra and relative retention times of the components in question by comparison to reference materials.

Target Compound Analysis

Target compound analysis uses key specific compounds to characterize an ignitable liquid. These target compounds are listed in Tables 3, 4, and 5.

Semi-quantitative ratios for the target compounds must be derived and compared against standards to ensure not only their presence but also that their chromatographic patterns match.

Target compound pattern recognition may be improved by the production of target compound chromatograms, which are graphical representations of semi-quantitative peak areas for the target compounds.

Target compound chromatograms for unknown samples are compared against those generated for reference samples. The same pattern matching criteria for mass chromatography apply to target compound chromatography.

Major peaks in the TIC not accounted for by one of the target compound types may be tentatively identified by searching their mass spectra against a suitable library.

This analysis provides useful information, however it should not be the sole basis for the identification of an ignitable liquid residue.

Interpretation

Compounds that comprise ignitable liquids consist of major types which have been organized into the following classification system or scheme, based on ASTM E1387 (Table 1). Note that the petroleum distillates have been broken down into light, medium, and heavy classifications:

Class Name	Peak Spread	Examples
Light Petroleum Distillate	C4 - C9	Petroleum ethers, pocket lighter fuels, some rubber cement solvents, Skelly solvents, V M & P Naphtha, some camping fuels
Gasoline	C4 - C12	All brands & grades of automotive gasoline, including gasohol
Medium petroleum Distillate	C8 - C13	Mineral spirits, some paint thinners, some charcoal starters, dry cleaning solvents, some torch fuels, some solvents for insecticides and polishes, some lamp oils
Heavy Petroleum Distillate	C9 - C20+	No. 1 & No. 2 fuel oil, kerosene, diesel fuel, some jet fuels, some charcoal starters, some solvents for insecticides
Miscellaneous	Variable	Single compounds, turpentines, enamel reducers, specialty mixtures that cannot be further classified into one of the categories below.
Oxygenated Solvents	Variable	Alcohols, esters, ketones
Isoparaffinic Products	Variable	Some charcoal starters, some copier toners, some aviation gasolines, some lamp oils, some specialty/industrial solvents
Normal Alkanes	Variable	Specialty products formulated from normal alkanes, some lamp oils, copier toner, carbonless paper
Aromatic Products	Variable	Specialty cleaning solvents, fuel additives, some paint and varnish removers, some automotive parts cleaners, xylenes, toluene based products
Naphthenic Paraffinic Products	Variable	Some charcoal starters, some paint thinners, some insecticide vehicles, some lamp oils, industrial solvents

The compounds that comprise ignitable liquids consist of six major types:

- Alkane (both normal and branched)
- Alkene
- Cycloparaffin
- Aromatic
- Polynuclear aromatic
- Oxygenates

Other compounds may be present, but are not considered significant for the purposes of this method.

Under the ignitable liquids scheme presented in Table 1, seven major classes of ignitable liquids may be identified by gas chromatography/mass spectrometry and/or ion profiling when recovered from fire debris. This is because a miscellaneous category is included for those ignitable liquids that do not fall into one of the major ignitable liquid classifications.

This method is intended to allow identified ignitable liquids to be characterized as belonging to one of these classifications. Distinguishing between examples within any class may be possible, but such further characterization is not within the scope of this method.

The products listed in Table 1 in the various classes are illustrations of known commercial uses these ignitable liquids have. These examples are not intended to be all-inclusive.

As can be noted, there are products found in multiple classifications such as "charcoal starters." Therefore, many of the examples can be preface by the word "some," as in "some charcoal starters."

Compounds of each type produce characteristic major ion fragments. These ions are listed in Table 2:

TABLE 2 - Major Ions Present in Mass Spectra of Common Flammable and Combustible Liquids*	
Compound Type	**m/e**
Alkane	**43, 57, 71, 85, 99**
Cycloalkane and alkene	**55, 69**
n-Alkylcyclohexanes	**82, 83**
Aromatic-alkylbenzenes	**91, 105, 119; 92, 106, 120**

Indanes	**117, 118; 131, 132**
Alkylnaphthalenes	**128, 142, 156, 170**
Alkylstyrenes	**104, 117, 118, 132, 146**
Alkylanthracenes	**178, 192, 206**
Alkylbiphenyls/acenaphthenes	**154, 168, 182, 196**
Monoterpenes	**93, 136**
Ketones	**43, 58, 72, 86**
Alcohols	**31, 45**
***R. Martin Smith,** Analytical Chemistry, Vol. 54, No. 13, November 1982, pp. 1399A-1409A.	

1. With the exception of the gasoline class, the major ignitable liquid classes may be divided into 3 subclasses based on boiling range: Light, Medium and Heavy.

2. Light product range: C_4-C_9, the majority of the pattern occurs in the range C_4-C_9, no major peaks exist above C_{11}.

3. Medium product range: C_8-C_{12}, narrow range products, the majority of the pattern occurs in the range C_8-C_{12}, no major peaks below C_7 or above C_{14}.

4. Heavy product range: C_9-C_{23}, typically broad range products, the majority of the pattern occurs in the range C_9-C_{23}, with a continuous pattern spanning at least 5 consecutive normal hydrocarbons, or ignitable liquid products starting above C_{11}.

5. It may be necessary to characterize a product as “light to medium,” or “medium to heavy,” when the carbon number range does not fit neatly into one of the above categories. In such instances, the carbon number range should be reported.

6. In order for an extract to be characterized as containing a particular class, the following minimum criteria must be met:

Criteria for the Identification of Gasoline

GENERAL: All brands of gasoline including gasohol. Pattern characterized by abundant aromatics in a specific pattern.

ALKANE: Petroleum distillate pattern comparable to that of a known standard with alkanes above C_9 present.

CYCLOALKANES: Not present in significant amounts.

AROMATIC: Petroleum distillate pattern comparable to that of known standards; 1-methyl-3-ethylbenzene (*n*-ethyltoluene), 1-methyl-4-ethylbenzene (*p*-ethyltoluene), 1, 3, 5-trimethylbenzene, 1-methyl-2-ethylbenzene (*o*-ethyltoluene), and 1, 2, 4-trimethylbenzene (pseudocumene) *must be present*; above C_7, the aromatic concentration is generally substantially higher than the alkane concentration.

CONDENSED RING AROMATIC: Pattern comparable to known standard is usually present, including naphthalene, 1- and 2- methylnaphthalenes. These compounds may be absent in some gasolines. Indan (dihydroindene) and methyl indans are usually present.

CAUTIONS: The mere presence of alkylbenzenes does not justify an identification of gasoline. These compounds must be present at approximately the same relative concentrations as are observed in samples of known gasoline. Many carpet samples that have been exposed to fire conditions contain these compounds in some concentrations. Benzene, toluene, ethylbenzene, xylenes, cumenes ethyltoluenes, and naphthalenes, which are present in gasoline, are also sometimes found in fire debris samples containing no foreign ignitable liquid residues. The presence of high levels of alkenes and oxygenates may indicate significant pyrolysis of the matrix and should make the recovery suspect. The presence of high levels of aromatics without the appropriate levels of alkanes may indicate an aromatic product.

Criteria for the Identification of Distillates

GENERAL: Traditional distillates and de-aromatized distillates; pattern typified by a Gaussian distribution of peaks with or without aromatic product present.

ALKANES: Abundant. Predominant n-alkanes present with isoparaffinic compounds present.

CYCLOALKANES: Present, less abundant than alkanes. Pattern varies by boiling range and peak spread.

AROMATICS: Always present in traditional medium and heavy distillates; less abundant than alkanes; pattern and abundance varies by boiling range and peak spread; may be present in light distillates. In some products, the aromatic composition may be significantly reduced or completely absent (de-aromatized).

CONDENSED RING AROMATICS: May be present based on boiling range and peak spread.

Criteria for the Identification of Isoparaffinic Products

GENERAL: Product comprised almost exclusively of branched chain aliphatic compounds (isoparaffins). The boiling range and pattern are dependent on the specific formulation.

ALKANES: Abundant. Pattern comparable to known isoparaffinic formulation. Characteristic isoparaffin product patterns present with no or insignificant levels of n-alkanes. The boiling range and component pattern are dependent on the specific formulation.

AROMATIC: Not present at significant concentrations.

CYCLOALKANES: Not present at significant concentrations. Note: Ions indicative of cycloparaffins are also present in smaller amounts in isoparaffinic compounds. "Cycloalkane" pattern representing isoalkanes may be present, but significantly less abundant than the alkane pattern.

CONDENSED RING AROMATICS: Not present.

Criteria for the Identification of Aromatic Products

GENERAL: Products comprised almost exclusively of aromatic and/or condensed ring aromatic compounds. The boiling range and pattern are dependent on the specific formulation.

ALKANE: Not present in significant amounts.

CYCLOALKANES: Not present in significant amounts.

AROMATIC: Abundant. Pattern comparable to known aromatic products. Pattern depends on formulation.

CONDENSED RING AROMATICS: May be present, pattern depends on formulation. Pattern comparable to known aromatic product.

NOTE: Light aromatic products may consist of single or few components. These compounds must be identified by both GC retention time and mass spectral identification**.**

Criteria for the Identification of Naphthenic Iso-Paraffinic Products

GENERAL: Products comprised mainly of branched chain (isoparaffinic) and cyclic (naphthenic) alkanes. The boiling range and pattern are dependent on the specific formulation.

ALKANES: Abundant. Normal alkanes may be notably absent or diminished. Depending on the feed stock (both de-aromatized distillates and dearomatized, deparaffinated distillates may be used), normal alkanes may be present, but at

diminished levels compared to distillate products. Pattern comparable to known naphthenic Iso-paraffinic products.

CYCLOALKANES: Abundant. Pattern comparable to known naphthenic Iso-paraffinic products.

AROMATICS: Not present in significant amounts.

CONDENSED RING AROMATICS: Not present in significant amounts.

Criteria for the Identification of Normal Alkane Products

GENERAL: Product comprised exclusively of normal alkanes. The boiling range and pattern are dependent on the specific formulation.

ALKANE: Normal alkane product pattern present with no isoparaffins or only minor levels of isoparaffins. The boiling range and pattern are dependent on the specific formulation.

CYCLOALKANES: Not significant.

AROMATIC: Not significant.

CONDENSED RING AROMATIC: Not significant.

NOTE: All major chromatographic peaks for this class must be identified by both GC retention time and mass spectral characteristics.

Criteria for the Identification of Oxygenated Solvents

GENERAL: Products containing major oxygenated components may include mixtures of oxygenated compounds and other compounds or products. Major oxygenated compounds present before C8; major compound(s) may include alcohols, esters, ketones. Other major compounds including toluene, xylene, and distillate formulations may also be present.

ALKANES: If in a mixture: may contain characteristic petroleum distillate pattern; pattern depends on formulation.

CYCLOALKANES: Pattern depends on formulation.

AROMATIC: Pattern depends on formulation.

CONDENSED RING AROMATIC: Not significant.

NOTE: All major oxygenated compounds must be identified by GC retention times and mass spectral characteristics.

CAUTION: The mere presence of oxygenated solvents such as alcohols or acetone does not necessarily indicate that a foreign ignitable liquid is present in the sample. There should be a large excess of the compound (at least one order

of magnitude above the other peaks in the chromatogram) before the analyst should consider the finding of an oxygenated product significant.

Miscellaneous/Other

No classification system is likely to describe all possible ignitable liquids. Flammable non-petroleum-based products, such as turpentine, do not fall into any of the categories. The other/miscellaneous category exists for these products.

Identification of ignitable liquid must be based on retention and mass spectra characteristics.

Factors Affecting Interpretation of Results

Pattern matching of mass chromatograms or target compound chromatograms rarely gives perfect correlation with reference material. In general, the unknown pattern (if positive) will be skewed toward less volatile compounds for weathered samples or skewed towards more volatile compounds for incompletely recovered samples. Compounds may be missing from either the light end or the heavy end, or both. Under certain conditions, selective loss of classes of compounds may result from microbiological degradation. Compounds may also be added to the pattern when the pyrolysis of materials at the fire scene yields target compounds or compounds of the same type as those being compared. All of these circumstances must be taken into account by the analyst during visual pattern evaluation. It is therefore imperative that the analyst have a sufficient library of commercially available ignitable liquids, in successive stages of evaporation. A library of extracts from common substrate materials containing no foreign ignitable liquids should also be maintained.

Interferences

Extraneous Components-Burned material from which the sample has been extracted usually contributes extraneous components to an extract. The amount and type of pyrolysis and combustion products formed during a fire depend on the substrate material and its fire history. They can consist of paraffinic, cycloparaffinic, aromatic, or condensed ring aromatic hydrocarbons, all of which will appear in the mass chromatograms. Only those pyrolysis products that are themselves target compounds listed in Tables 3, 4, and 5 will appear on the target compound chromatograms. The presence of these extraneous product components is acceptable when sufficient ignitable liquid product compounds remain to allow proper classification of the sample. When the pattern becomes overwhelmed by extraneous components, identification is not possible by this method.

Extracts that meet the criteria for heavy petroleum distillates should be reviewed carefully for "extraneous components" that elute near n-alkanes and are the result of polyolefin or high molecular weight hydrocarbon (asphalt) decomposition. Peaks representing the corresponding 1-alkene or 1, (n-1) diene, and having a concentration near the concentration (within 1/2 an order of magnitude) of the alkane, should be considered as indicating the presence of polyolefin products rather than fuel oil products. Polyolefin decomposition products typically do not exhibit the same pattern of branched alkanes as fuel oils.

Missing Components-Exposure of the ignitable liquid to heat usually results in the preferential loss of lighter components, thereby enhancing the chromatographic pattern at the heavy end. Some sample preparation techniques may result in the preferential recovery of either the lighter or heavier components, resulting in the "loss" in the opposite end. Neither of these factors will cause the selective loss of intermediate components. The unexplainable absence of components from the middle of a pattern is generally sufficient grounds for a negative finding. Possible explanations for missing intermediate compounds include low sample concentration (compound below detection limit), compound did not meet target compound identification criteria (due to distortion of the mass spectrum by a co-eluting extraneous compound), and, in rare cases, selective loss due to digestion by microbes. Any such explanation for loss of compounds in the middle of a pattern must be scientifically supportable, and efforts should be made, if possible, to retrieve evidence of their existence from the data file or by reanalyzing the sample.

The presence of small quantities of some compounds common to a particular class of ignitable liquid product does not necessarily indicate the presence of that liquid in the debris at the time of the fire.

For example, the pyrolyzates of aromatic-containing polymers may include toluene and xylenes. The pyrolyzates of asphalt and polyolefin plastic may include a homologous series of normal alkanes.

Certain ignitable liquid components may be found in some substrates at the fire scene. Examples include:

- normal alkane products found in linoleum and in carbonless paper forms
- distillates found in some printed materials
- certain solvents used in some adhesives and coatings.

If there is suspicion that an ignitable liquid found might be indigenous to the substrate, the testing of an appropriate comparison sample, if available, may aid in determining whether or not an ignitable liquid is foreign to the substrate.

Factors Affecting Interpretation

1. Some samples are weathered (partially evaporated) when received
2. Distortion of the total ion chromatogram because of components being trapped in the matrix more firmly than others
3. Components being lost because of bacterial action
4. Contamination due to either low concentrations of sample or high concentration of interfering components
5. Mixtures of ignitable liquids
6. A complete spectrum of one substance being included in another
7. Weathered samples of one sample resembling weathered samples of another

TABLE 3 Gasoline Target Compounds / Ions	
1. 1,3,5-Trimethylbenzene	108-67-8
2. 1,2,4-Trimethylbenzene	95-36-3
3. 1,2,3-Trimethylbenzene	526-73-8
4. Indane	496-11-7
5. 1,2,4,5-Tetramethylbenzene	95-93-2
6. 1,2,3,5-Tetramethylbenzene	527-53-7
7. 5-Methylindane	874-35-1
8. 4-Methylindane	824-22-6
9. Dodecane	112-40-3
10. 4,7-Dimethylindane	6682-71-9
11. 2-Methylnaphthalene	91-57-6
12. 1-1-Methylnaphthalene	90-12-0
13. Ethylnaphthalenes (mixed)	1127-76-0
14. 1,3-Dimethylnaphthalene	575-41-7
15. 2,3-Dimethylnaphthalene	581-40-8

TABLE 4 Medium Petroleum Distillate (MPD) Target Compounds / Ions	
1. Nonane	111-84-2
2. Propylcyclohexane	1678-92-8
3. 1,3,5-Trimethylbenzene	108-67-8
4. 1,2,4-Trimethylbenzene	95-36-3
5. Decane	124-18-5
6. 1,2,3-Trimethylbenzene	526-73-8
7. n-Butylcyclohexane	1678-93-9
8. Trans-decalin	493-02-7
9. Undecane	1120-21-4
10. 1,2,3,5-Tetramethylbenzene	527-53-7
11. n-Pentylcyclohexane	4292-92-6
12. Dodecane	112-40-3
13. n-Hexylcyclohexane	4292-75-5

TABLE 5 Heavy Petroleum Distillate (HPD) Target Compounds / Ions	
1. Decane	124-18-5
2. n-Butylcyclohexane	1678-93-9
3. Trans-decalin	493-02-7
4. Undecane	1120-21-4
5. 1,2,3,5-Tetramethylbenzene	527-53-7
6. n-Pentylcyclohexane	4292-92-6
7. Dodecane	112-40-3
8. n-Hexylcyclohexane	4292-75-5
9. 2-Methylnaphthalene	91-57-6
10. 1-Methylnaphthalene	90-12-0
11. Tridecane	629-50-5
12. n-Heptylcyclohexane	
13. 1,3-Dimethylnaphthalene	575-41-7
14. Tetradecane	629-59-4
15. n-Octylcyclohexane	1795-15-9
16. 2,3,5-Trimethylnaphthalene	2245-38-7

TABLE 5 Heavy Petroleum Distillate (HPD) Target Compounds / Ions	
17. Pentadecane	629-62-9
18. n-Nonylcyclohexane	2883-02-5
19. Hexadecane	544-76-3
20. Heptadecane	629-78-7
21. Pristane	1921-70-6
22. Octadecane	593-45-3
23. Phytane	638-36-8
24. Nonadecane	629-92-5
25. Eicosane	112-95-8
26. Heneicosane	629-94-7

Case File

The bench notes will contain:

- Fire Debris Worksheet.
- The analyst's name and laboratory case number on each page of file.
- A description of each item of evidence and its packaging.
- A copy of the instrument and column parameters.
- Documentation of instrument tune within one month.
- Extraction method used (e.g. passive headspace) for each item.
- Extraction details such as oven temperature and adsorption time.
- Lot number of activated charcoal strips (when used).
- Standard Accelerant Mixture (5% SAM) spectra.
- Blank spectra from activated charcoal strip spectra (with passive headspace), elution solvent, and/or empty fire debris container.
- Test spectra for all reported evidence items.
- Extracted ion chromatograms when utilized for interpretation.
- Blank spectra for each evidence item analyzed by GC/MS.
- Total and extracted ion chromatograms for standards utilized for comparison.
- Conclusions reached from analysis.
- Disposition of adsorbed but unused charcoal strips
- Dates that analysis began and was completed.

The LIMS case file will also contain:

- Request for Laboratory Services
- Complete chain of custody information for all evidence.

Reports

The REPORT will contain:

- Agency case number
- Submitting Agency
- Name of person submitting evidence
- Item numbers and descriptions of submitted evidence
- Results of laboratory analysis

The report may contain examples of commercial products and/or substrates that might contain the ignitable liquid identified. The conclusion should give the scientist's opinion as to whether or not an ignitable liquid was identified in the sample. If a negative result was obtained, a disclaimer may be offered to the effect that it does not preclude the possibility that ignitable liquids were present at the fire scene. In the case of a positive report, it may be appropriate to add a disclaimer to the effect that the identification of an ignitable liquid residue in a fire scene does not necessarily lead to the conclusion that a fire was incendiary in nature. Terms such as hydrocarbons, consistent with, in the boiling range of, similar to, or characteristic of will not be used without additional explanation or a positive identification of the product. Results may be reported using the classification presented in Table I or as a light, medium, or heavy petroleum product. See the explanation following Table I for further guidance. Ignitable liquids are not necessarily reported as but may be classified by the guidelines appearing in ASTM E 1618-10.

References

GC-MS Guide to Ignitable Liquids, Reta Newman, CRC Press, 1998.

ASTM Standard E 1386-00: Standard Practice for Separation and Concentration of Ignitable Liquid Residue from Fire Debris Samples by Solvent Extraction.

ASTM Standard E 1387-01: Standard Test Method for Ignitable Liquid Residues in Fire Debris Samples by Gas Chromatography.

ASTM Standard E 1388-00: Standard Practice for Sampling of Headspace Vapors from Fire Debris Samples.

ASTM Standard E 1412-00: Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration.

ASTM Standard E 1618-10: Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography/Mass Spectrometry.

APPENDIX A Abbreviations

ASTM: American Society of Testing Materials

GC: gas chromatograph

HP: Hewlett Packard

EIC: extracted ion chromatogram

m/e: mass per charge

µL: microliter

MS: mass spectrometer

TIC: total ion chromatogram

TCC: target compound chromatogram

SAM: Standard Accelerant Mixture

APPENDIX B Review Checklist

LAB NUMBER:
ANALYST:

TECHNICAL REVIEW **Analyst:** **Date:**

Yes	N/A	
☐	☐	The container and a brief description of its contents are documented.
☐	☐	The sample preparation and analytical technique performed is recorded.
☐	☐	The GC conditions are documented.
☐	☐	The GCMS tune has been done as needed or within approximately one month.
☐	☐	The carbon strip lot #, oven temperature, and the adsorption time are recorded.
☐	☐	The 5% SAM and solvent blank from the time period of the analysis are included.
☐	☐	For each GC/MS spectra, a blank is included in the notes.
☐	☐	For each identified ignitable liquid, a standard (or use of the SAM) is included..
☐	☐	Bench notes include an explanation of identifying characteristics (if applicable).
☐	☐	The analyst's conclusions in the report are supported by documentation in the bench notes.
☐	☐	The disposition of the unused carbon strip portion is documented.
☐	☐	The worksheet indicates the dates that analysis was started and completed.
☐	☐	All of the bench notes, scanned data and worksheet are in LIMS.

ADMINISTRATIVE REVIEW **Analyst:** **Date:**

Yes	N/A	
☐	☐	The requesting agency, agency number, & lab number agree between the lab request and the lab report.
☐	☐	The correct submitting officer or whom to reply to appears on the report.
☐	☐	Item numbers & descriptions agree between LIMS, bench notes, spectra & report.
☐	☐	Grammar and punctuation are correct.
☐	☐	The report is signed by the analyst.
☐	☐	All of the bench notes and attached/scanned documents are present in LIMS.
☐	☐	The report is referred for release to the submitting agency.
☐	☐	The electronic chain-of-custody in LIMS agrees with the worksheet and report.

APPENDIX C ASTM Documents

See the laboratory network:

I:\Uncontrolled Documents\Section Shares\Fire Debris_Share\ASTM Documents

APPENDIX D Uncertainty of Measurement

Since fire debris analysis is a qualitative testing method, the term *measurement* does not apply.

Appendix E Revision History

Section(s) Revised	Date	Issuing Authority
Overview, p. 3, ASTM standard E1618-01 changed to E 1618-10.	12-13-2011	Jane Booth, Supervisor
Table 1, p. 12, Notation that petroleum distillates class from ASTM E 1387 broken down into light, medium, and heavy categories.	12-13-2011	Jane Booth, Supervisor
Interferences p. 20, bullet points for sources of ignitable components from a fire scene.	12-13-2011	Jane Booth, Supervisor
Case File, p. 23, addition of extracted ion chromatograms in bench notes.	12-13-2011	Jane Booth, Supervisor
Case File, p. 23, addition of total and extracted ion chromatograms of any standards utilized for comparison.	12-13-2011	Jane Booth, Supervisor
Reports, p. 24, changed ASTM E 1618-01 to E 1618-10.	12-13-2011	Jane Booth, Supervisor
Appendix B, Review Checklist, p. 27 revised.	12-13-2011	Jane Booth, Supervisor
Appendix D, Uncertainty of Measurement, p. 28, not applicable to fire debris analysis	12-13-2011	Jane Booth, Supervisor